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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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National food safety standards -- Food additives -- Methanol

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(English Translation)

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1 Scope

This standard applies to coal, natural gas, light oil, heavy oil as raw material for the production of food additives methanol.

2.1 Molecular formula

CH₃OH

2.2 Relative molecular mass

32.04 (according to 2007 International relative atomic mass)

3.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color and colorless State transparent liquid Take appropriate sample, placed in a clean, dry colorimetric tube, in the self Under the light, observe the color and state

3.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Methanol content, w /% $\geq$ 99.5 Appendix A, A.4 Moisture, w /% $\leq$ 0.1 GB 5009.3 Karl Fischer method Carbonyl compounds (in formaldehyde), w /% $\leq$ 0.010 GB/T 6324.5-2008 4.2a Distillation range/°C 64.5 ~ 65.5 GB/T 7534 Evaporation

residue/(mg/100mL) $\leq$ 3 GB/T 6324.2

Table 2 (continued)

Item Index Test Method Acidity (in formic acid)/(mg/kg) $\leq$ 15 Appendix A A.5 Alkalinity (in ammonia)/(mg/kg) $\leq$ 3 Appendix A A.6 Lead (Pb)/(mg/kg) $\leq$ 2 GB 5009.12 a measurement wavelength of 430nm.

Appendix A

Testing method

A.1 Warning

The test procedure specifies that some of the test procedures may lead to dangerous conditions and that the operator should take appropriate safety and protective measures.

A.2 General provisions Unless otherwise stated, only the reagents identified as analytical reagents and the tertiary water specified in GB/T 6682-2008 are used in the analysis. test Method used in the standard titration solution, impurity determination standard solution, preparation and products, without any other requirements, according to GB/T 601, GB/T 602 and GB/T 603; the solution used in the absence of the solvent, refers to the aqueous solution.

A.3 Identification test A.3.1 Solubility Soluble in water, ether and ethanol.

A.3.2 Refractive index According to the provisions of GB/T 614 determination. Refractive index n_D^{20} should be. 1.328 ~ 1.330.

A.3.3 Density

According to the provisions of GB/T 4472 to determine. The density (ρ_{20}) should be 0.790 g/cm³ to 0.793 g/cm³.

A.3.4 Boiling point

According to the method specified in GB/T 7534. The boiling point should be about 65 ° C.

A.4 Determination of methanol content A.4.1 Methodological Summary Under the selected working conditions, the samples were vaporized through the column, the components were separated and detected with a hydrogen flame ionization detector. use Determination of Methanol Content by Area Normalization Method.

A.4.2 Reagents and materials Calibration solution (mixed with methanol and 1,4-dioxane). 1 250.

A.4.3 Instruments and equipment A.4.3.1 Gas chromatograph. equipped with flame ionization detector, the machine sensitivity and stability in line with GB/T 9722 in the relevant provisions.

A.4.3.2 Recorder. Chromatographic data processor or chromatographic workstation.

A.4.3.3 Sampler. Microinjector.

A.4.4 Columns and Typical Chromatographic Operating Conditions the recommended column and typical chromatographic operating conditions are given in Table A.1. Other columns that can meet the analytical requirements and chromatographic operating conditions are also available use.

Table A.1 Recommended Columns and Typical Chromatographic Operating Conditions

Column

Column length 1.8m, column diameter 4mm, filler 105 μ m ~ 120 μ m benzene Ethylene - divinylbenzene polar monomer copolymer Column temperature/ $^{\circ}$ C 160 Vaporization chamber temperature/ $^{\circ}$ C.200 Detector temperature/ $^{\circ}$ C 210 Carrier gas (nitrogen) Flow rate/(mL/min) 25 Injection volume/ μ L 5 ~ 10 A.4.5 Analysis steps the temperature of the mild (or) carrier gas of the regulator column is such that the retention time of the methanol is about 5 min to 7 min. Adjust the detector to make 8 μ L of the calibration solution the resulting peaks are in the middle of the spectral range. Injection of 5 μ L ~ 10 μ L sample, to obtain the chromatogram, with the area normalization method Methanol content.

A.4.6 Calculation of results the mass fraction w_1 of methanol is calculated according to formula (A.1) $w_1 = A_1 / \sum A_i \times 100\%$ (A.1) where.

A_1 --- the peak area of methanol;

$\sum A_i$ --- the sum of the peak areas of all components.

A.5 Determination of acidity (in form formic acid) A.5.1 Reagents and materials A.5.1.1 Ethanol.

A.5.1.2 Sodium hydroxide standard titration solution. $c(\text{NaOH}) = 0.02 \text{ mol/L}$.

A.5.1.3 phenolphthalein indicator solution. 10g/L.

A.5.2 Analysis steps 10 mL of ethanol and 25 mL of water were mixed by adding 0.5 mL of phenolphthalein indicator solution, titrated with sodium hydroxide titration solution titrated to the first the pink appears and remains at least 30s. Add 19 mL (about 15 g) of the sample, mix and titrate the titration solution with sodium hydroxide standard until Times appear pink. Sodium hydroxide standard titration solution should be used in the amount of not more than 0.25mL.

A.6 Determination of alkalinity (in ammonia) A.6.1 Reagents and materials A.6.1.1 Sulfuric acid standard titration solution. $c(1/2\text{H}_2\text{SO}_4) = 0.02 \text{ mol/L}$.

A.6.1.2 methyl red indicator solution. 1g/L.

A.6.2 Analysis steps Add 1 drop of methyl red indicator to 25 mL of water and titrate to red

with standard titration solution of sulfuric acid. Add 29 mL (approx.

22.5 g) sample, mix well. Titration with sulfuric acid standard titration solution to red again, sulfuric acid standard titration solution should not be used at 0.2 mL.